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Physics Procedia 11 (2011) 122–125

Physics Procedia

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Proceedings of APAC-SILICIDE 2010

Thermal Process of Iron Silicides prepared by Magnetron sputtering

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Abstract

The effects of annealing temperature and duration on the formation of iron silicides prepared by magnetron sputtering (MS) are investigated. The Fe/Si structures were annealed at 1073 K-1223 K for 6-18 hours. The results show that 1143 K is a critical temperature to the formation of β-$FeSi_2$ from Fe/Si structures deposited at room temperature. The single phase semiconducting β-$FeSi_2$ can be obtained after annealing at 1173 K for 12-18 hours. The SEM images indicate that both annealing temperature and duration have obvious effects on microstructures of the film. When annealing time is more than 15 hours, the crystallinity of β-$FeSi_2$ improves and the distribution of grains becomes homogeneous. Most of the grains of β-$FeSi_2$ grow toward substrate and

Keywords: Magnetron sputtering; Semiconducting beta iron- disilicide; Thermal treating process; Crystal structure and microstructure.

1. Introduction

In a solid-state reaction, iron and silicon can form various compounds with different phases which can coexist [1]. Among these compounds, semiconducting iron disilicide β-$FeSi_2$ with orthorhombic structure has recently attracted much attention as a suitable material for Si-based optoelectronic devices [1-4]. Growth techniques such as MBE and IBS have been investigated extensively [5-9], however, the study for thermal process of iron silicide prepared by magnetron sputtering (MS) is relatively few [10-12]. In this work, we tried to find the critical thermal process condition by investigating the effect of annealing temperature and duration on the formation of iron silicides prepared by MS.

2. Experiments

Fe films about 100 nm were deposited by JGP 560 magnetron sputtering system onto Si (100) substrates (p-type, 7-13 $\Omega \cdot cm$). The Si wafers were ultrasonically cleaned for 10 minutes in acetone, ethanol and deionized water one after another before mounting in the chamber. The purity of Fe target was 99.95% and the base pressure was 2×10^{-5} Pa before sputtering. The sputtering power is 110 W and the Ar pressure was 2.0 Pa and the substrates were

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doi:10.1016/j.phpro.2011.01.019

held at room temperature during the sputtering process. Subsequent annealing was performed in the temperature range of 1073 K-1223 K for 6-18 hours in a vacuum furnace. The formation of iron silicides is clarified by X-ray diffraction (XRD, D/MAX-2200, Cu-Kα) and the surface microstructure is characterized by scanning electron microscope (SEM, HITACHI S3400) at an acceleration voltage of 20 kV

3. Results and discussion

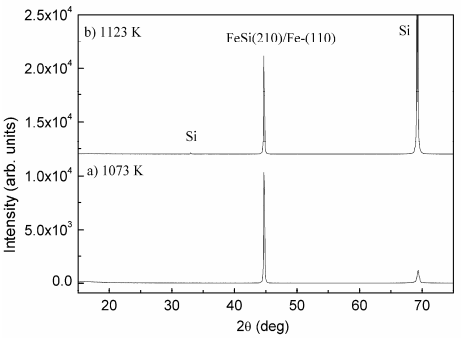

Fig.1 XRD patterns for Fe/Si layers annealed at 1073 K and 1123 K for 15 hours.

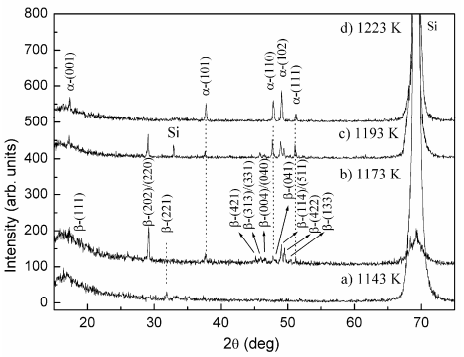

Fig.2 XRD patterns for Fe/Si layers annealed at 1143 K to 1223 K for 15 hours.

Fig. 1 and 2 are the XRD patterns for Fe/Si layers deposited at room temperature and annealed at 1073 K to 1223 K for 15 hours. The results show that 1143 K is a critical temperature, the intensity of diffraction peak of the samples annealed at lower temperature is far higher than that annealed at high temperatures, the counts of the highest peak in former is104 but it is only 102 in later. In former, there is only one peak at 44.986º appeared which may be contributed by FeSi (210) or Fe (110). Considering it is the result of annealed for 15 hours, it should be FeSi (210) but Fe (110) and the results is confirmed by energy dispersion spectra (EDS). β-$FeSi_2$ just begin to form at 1143 K and the single phase β-$FeSi_2$ can be obtained at 1173 K, whereas β-$FeSi_2$ and α-$FeSi_2$ are coexist together at 1193 K and all silicides transformed into high temperature phase α-$FeSi_2$ when annealing temperature goes up to 1223 K.

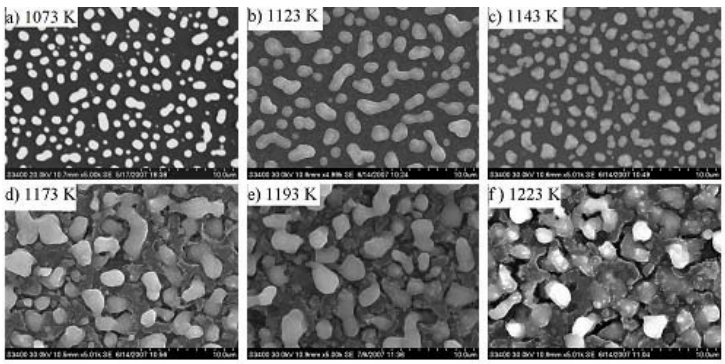

Fig.3 SEM images for Fe/Si layers deposited at room temperature and annealed at 1073 K to 1223 K for 15 hours

Fig.3 is the SEM images for Fe/Si layers deposited at room temperature and annealed at 1073 K to 1223 K for 15 hours. To microstructure of the film, 1143 K is also a boundary, Fe or FeSi form separate island grains on the surface of Si matrix when anneal temperature is lower than 1143 K, once the anneal temperature is over than 1143 K, β-$FeSi_2$ begin to grow by consuming FeSi, consequently the grain size of FeSi decrease and begin to melt which leads the boundary of grains becomes to be mistiness and loses the shape character of polyhedron, the crystal of β-$FeSi_2$ has completed and embedded themselves into Si matrix when anneal temperature goes up to 1173 K. The surface morphology of the film is almost no change when the anneal temperature increases further, but the crack can be observed on the surface of the film due to the high temperature and long time of thermal treating.

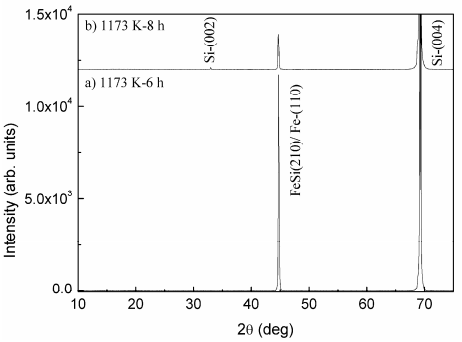

Fig.4XRD patterns for Fe/Si layers deposited at room temperature and annealed at 1173 K for 6-8 hours.

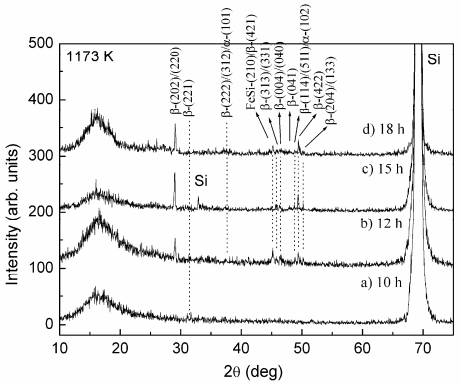

Fig.5 XRD patterns for Fe/Si layers deposited at room temperature and annealed at 1173 K for 10-18 hours.

Fig.4 and Fig.5 are the XRD patterns for Fe/Si layers deposited at room temperature and annealed at 1173 K for 6-18 hours. The effect of anneal leads the Fe film to be crystal and grow firstly, then Fe and Si atoms begin to react and form intermediate phase silicide FeSi, FeSi reacts with Si atoms comes from substrate and forms β-$FeSi_2$ when the anneal time goes up to 10 hours, after that, the peak numbers of β-$FeSi_2$ increase and peak intensity enhances when anneal time increases up to 12 hours, finally the peak of β-$FeSi_2$ is consistent with PDF card #20-0532, the intensity of the peaks arrives the maximum as the anneal time goes up to 1173 K, the peak intensity decreases if the anneal time increases further.

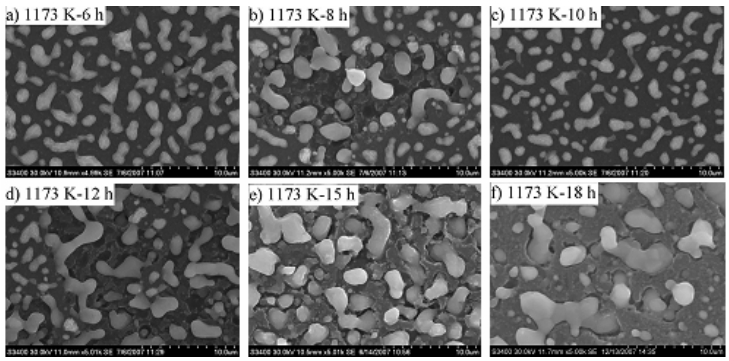

Fig.6 SEM images for Fe/Si layers deposited at room temperature and annealed at 1173 K for 6-15 hours

Fig.6 is the SEM images for Fe/Si layers deposited at room temperature and annealed at 1173 K for 6-18 hours. The anneal time has obvious effects on microstructures of the film, too. After anneal at 1173 K for 6 hours, the grains on the surface appear to be separate islands, the boundary of grains looks to be mistiness, it is the first step for the formation of Fe-Si compounds, the intermediate phase FeSi forms. The crystal of FeSi is complete and the boundary of grain becomes clear when anneal time increases to 8 hours. When the anneal time increases to 10 hours, the grain size become small and the surface of the grains becomes mistiness again, this is the second step of formation of Fe-Si compounds, β-$FeSi_2$ grows by consuming FeSi. When the anneal time increase to 12 hours, the boundary of grains becomes clear again which shows the crystal of β-$FeSi_2$ to be completed. The anneal time continuous increase up to 15 hours, the crystallinity of β-$FeSi_2$ improves, the size of grains increases and the distributing of grains become homogeneous. Most of the grains grow toward substrate and embed themselves into Si matrix.

4. Conclusion

The effect of annealing temperature and annealing time on the formation of Fe-Si compounds prepared by magnetron sputtering is investigated. The Fe/Si structures prepared by magnetron sputtering were annealed at 1073 K-1223 K for 6-18 hours. XRD results indicate that the single phase semiconducting β-$FeSi_2$ can be obtained for Fe/Si structures deposited at room temperature and subsequently annealed at 1173 K for 12-18 hours. The SEM images indicates that both the anneal temperature and anneal time have obvious effects on microstructures of the film. When anneal time is more than 15 hours, the crystalline of β-$FeSi_2$ improves and the distribution of grains becomes homogeneous. Most of the grains of β-$FeSi_2$ grow toward substrate and embed themselves into Si matrix.

Acknowledgements

This research was supportedThis research was supported by the National Science Foundation of China under grant number 60566001, the International Cooperation Research Program of the Ministry of Science and Technology, China [2008DFA52210], the Natural Science program [J(2008)2002] and the International Cooperation Research Program [G(2009)700113] of Guizhou Province, China, and the talent program of Guizhou University [(2009)002].

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